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TITANIUM DIOXIDE PARTICLES HAVING BENEFICIAL PROPERTIES AND METHOD FOR PRODUCING THE SAME

[0001]

Field of the Invention

The present invention relates to titanium dioxide particles having beneficial properties such as highly selective shielding of thermal infrared radiation and highly spreadable property. It also relates to a method for producing such titanium dioxide particles.

[0002]

Background of the Invention

Generally infrared radiation refers to electromagnetic radiation above the wavelength range of 0.76-0.83 μm of visible light reaching the wavelength of several millimeters. The solar radiation reaching the global surface comprises approximately 2 % of ultraviolet(UV), 48% of visible and 50% of infrared(IR) radiation. Most of IR are converted to thermal energy.

[0003]

Titanium dioxide(TiO_2) particles having a primary particle size range from about 0.2 μm to

about 0.4 μm have a high refractive index and a high reflectivity to visible light, and thus have a high hiding power which makes the particles useful as white pigment for use in the production of paints, printing inks, plastic molding compounds, cosmetic preparations and so on.

[0004]

TiO_2 microparticles having a primary particle size of less than 0.1 μm exhibit a low reflectivity and are transparent to visible light. However, they exhibit high shielding in the UV wavelength range that makes them useful as UV blocker in cosmetic and other preparations.

[0005]

Because of their high reflectivity to visible light, the TiO_2 particles having a primary particle size from about 0.2 μm to about 0.4 μm used as white pigment are known to shield the visible wavelength range of the solar radiation. Therefore, they also have some heat shielding effect against the solar radiation. By "heat shielding effect" as used herein, it is meant the ability of preventing the elevation of internal temperature of an object exposed to the solar radiation by scattering the solar radiation on the surface thereof. In order to further

increase the heat shielding effect, it would be required for TiO_2 particles to further increase the particle size. TiO_2 particles dedicated to shielding the thermal IR have not been developed to the best of our knowledge.

[0006]

TiO₂ particles having different particle size range and optical properties from those of pigment grade and UV blocker TiO_2 particles are known. For example, JP-A-6018807 discloses a makeup cosmetic preparation comprising TiO_2 having a mean particle size in the range between 0.4 and 20 μm. This preparation is alleged to have aesthetically natural finish and improved extendability onto the skin. JP-A-09221411 discloses TiO_2 having a mean particle size greater than 0.10 μm and less than 0.14 μm. It is alleged that the TiO_2 in the above particle size has, when formulated in cosmetic preparations, a suitable level of hiding power while retaining UV blocking effect so as to impart aethetically natural finish free of pale appearance. JP-A-11158036 and JP-A-2000327518 disclose primary TiO_2 particles of 0.01 to 0.15 μm size that have been agglomerated into secondary particles of 0.6 to 2.0μm size. They are formulated in cosmetic preparations in

conjuction with plastic microbeads such as silione microbeads. The agglomerate is said to be transparent to visible light while retaining a large extent of the UV blocking effect of the primary particles without pale appearance.

[0007]

TiO_2 of the white pigment grade has been adjusted to a primary particle size for efficiently scattering visible light in the wavelength range between 0.4 μm and 0.8 μm. Consequently, the ability thereof to shield thermal IR in the wavelength range higher than visible light is considered to be low in practice for IR shielding applications. If the IR shielding effect of TiO_2 can be increased significantly, it would find use as a thermal IR shielding material in a variety of compositions including paint compositions to be applied on houses and buildings, ships, automotives, household electrical and electronic equipment, drink cans, roads and the like to prevent them from exposing to an elevated temperature. Such a thermal IR shielding material would find use in cosmetic preparations for the prevention of elevated skin temperature.

[0008]

In order to effectively shield thermal IR of the wavelength range between 0.8 μm and 3.0 μm, the TiO_2 particles need to have a wide distribution of the size of individual primary particles in the range between 0.4 μm and 1.5 μm in theory.

[0009]

TiO_2 particles of pigment grade or UV blocker grade have been used in cosmetic preparations such as liquid foundations or powders as describe above. It is important for these preparations to have a high spreadability when applying to or spreading on the skin. The tactile feeling of cosmetic preparations containing TiO_2 of the pigment grade or UV blocker grade is not satisfactory due to TiO_2 particles themselves and the TiO_2 particles are often formulated in conjuction with a spreadability improver.

[0010]

One approach for producing TiO_2 having larger particle size than the pigment grade TiO_2 in the existing plant for the sulfuric acid process would be to calcine hydrated TiO_2 at a temperature higher than the temperature at which hydrated TiO_2 is calcined to produce the pigment grade TiO_2. However, this process gives particles which are hardly

dispersible in a medium as fine particles due to increased fraction of fused fine particles. Moreover, the growth of the particles in the direction of miner axis during the calcination is not sufficient compared to the direction of major axis resulting in generally rod-like particles having decreased scattering efficiency.

[0011]

JP-B-50036440 discloses a process for producing a pigment grade TiO_2 comprising blending hydrated TiO_2 produced by the hydrolysis of titanyl sulfate with certain amounts of zinc sulfate and potassium sulfate, and calcining the blend at a temperature between 700℃ and 1,000℃. The resulting TiO_2 contains a large amount of needle crystals of TiO_2, and the optical properties and primary particle size thereof are not different from the TiO_2 of pigment grade.

[0012]

Brief Summary of the Invention

Accordingly, it is a principal object of the present invention to provide TiO_2 particles having a different primary particle size from that of the pigment grade or UV blocker grade and a number of beneficial properties including the ability of

selectively shielding IR radiation and the ability of improving the spreadability of cosmetic preparations. Another object is to provide a process for producing the above TiO_2 particles.

[0013]

The above and other objects of the present invention may be achieved by providing TiO_2 particles having a primary particle size between 0.5 and 2.0 μm and a reflectivity to the visible light less than 95%.

[0014]

According to another aspect of the present invention, the above TiO_2 particles may be produced by blending hydrated TiO_2, based on the TiO_2 content thereof, with 0.1 to 0.5% by weight of an aluminum compound calculated as Al_2O_3, 0.2 to 1.0% by weight of zinc compound calculated as ZnO, and 0.1 to 0.5% by weight of a potassium compound calculated as K_2CO_3; and calcining the resulting blend at a temperature between 900℃ and 1,100℃. The TiO_2 particles thus produced contain at least 0.05 to 0.4% by weight of Al_2O_3 and 0.05 to 0.5% by weight of ZnO, the most part thereof, namely 0.05 to 0.3 wt.% of Al_2O_3 and 0.05 to 0.5 wt.% of ZnO being present in the crystalline lattice.

[0015]

The TiO_2 particles of the present invention may be incorporated into paints, printing inks or plastic molding compounds for shielding the thermal IR radiation. Due to relatively low reflectivity to the visible light, the TiO_2 particle of the present invention may be used in conjuction with conventional color pigments without whitening to impart a colored paint film with IR shielding effect. When incorporated in cosmetic preparations, the TiO_2 particles of the present invention may improve the spreadability in particular onto the skin compared to the pigment or UV blocker grade TiO_2. The TiO_2 particles of the present invention do not generate bluish luminescence observed in the TiO_2 white pigment due to decreased reflectivity to the visible light.

[0016]

Brief Description of the Accompanying Drawings

Fig. 1 is a graph showing a transmission curve in the IR range of the film containing TiO_2 produced in Example 1.

Fig. 2 is a graph showing a transmission curve in the IR range of the film containing TiO_2 produced in Example 2.

Fig. 3 is a graph showing a transmission curve in the IR range of the film containing a commercial TiO_2 pigment.

Fig. 4 is a graph showing a transmission curve in the IR range of the film containing zinc titanate rather than TiO_2.

Fig. 5 is a graph showing a reflection curve in the visible range of TiO_2 produced in Example 1.

Fig. 6 is a graph showing a reflection curve in the visible range of TiO_2 produced in Example 2.

Fig. 7 is a graph showing a reflection curve in the visible range of a commercial TiO_2 pigment.

[0017]

Detailed Description of the preferred Embodiments

According to the present invention, the TiO_2 particles having a primary particle size between 0.5 and 2.0 μm are produced starting from hydrated TiO_2. Blended with the hydrated TiO_2 are, based on the TiO_2 content thereof, an aluminum compound in an amount corresponding to 0.1 to 0.5% by weight calculated as Al_2O_3, a potassium compound in an amount corresponding to 0.1 to 0.5% by weight calculated as K_2CO_3, and a zinc compound in an amount corresponding to 0.2 to 1.0% by weight calculated as ZnO. After drying, the resulting blend

is calcined at a temperature between 900℃ and 1,100℃.

[0018]

The starting hydrated TiO_2 may be produced, for example, by treating titanium-containing ore such as ilmenite or rutile with sulfuric or hydrochloric acid to remove impurities, and then adding water or an oxidizing agent to the resultant solution to precipitate hydrated TiO_2. Hydrated TiO_2 may be produced by hydrolyzing a titanium alkoxide. Metatitanic acid produced as an intermediate of TiO_2 pigment in the commercial sulfuric acid process is a preferred starting material.

[0019]

Any aluminum compound may be added to hydrated TiO_2 provided that it does not adversely affects the desired properties of TiO_2 of the present invention. A water soluble aluminum salt such as the sulfate or chloride is preferred although the oxide or hydrated oxide may also be used. The amount of the aluminum compound to be added calculated as Al_2O_3 ranges between 0.1 and 0.5% by weight relative to the TiO_2 content of hydrated TiO_2.

[0020]

Any potassium compound may also used.

Examples thereof include the hydroxide, carbonate or chloride. The amount of potassium compound to be added ranges between 0.2 and 0.5% by weight calculated as K_2CO_3 relative to the TiO_2 content of hydrated TiO_2. In the absence or presence in only trace amounts of the potassium compound, a large portion of individual primary particles will be firmly fused together so that dispersion into individual primary particles will become difficult and the desired IR shielding effect will decrease. Conversely, excessive addition of the potassium compound will result in the formation of rod-like particles with decreased IR shielding effect or decreased conversion to rutile crystals in the desired particle size.

[0021]

Any zinc compound may also be added to hydrated TiO_2. Preferred examples thereof include the oxide, sulfate or chloride. The amount of the zinc compound to be added ranges between 0.1 and 1.0% by weight calculated as ZnO relative to the TiO_2 content of hydrated TiO_2. In the absence or presence in only trace amounts of the zinc compound, the proportion of rod-like particles with decreased IR shielding effect will increase.

[0022]

Besides, dispersion of the product into individul primary particles will become difficult again due to firm fusion of primary particles together since a higher temperature is required for growing fine particles to the desired particle size in the presence of the zinc compound in excess. As is known in the art, the zinc compound reacts with TiO_2 at an elevated temperature to produce zinc titanate having a refractive index lower than TiO_2 pigment and hence the larger in the proportion of zinc titanate in the product the lower in the IR shielding effect. Excessive addition of the zinc compound is not preferable also for this reason.

[0023]

The addition of aluminum, zinc and potassium compounds to hydrated TiO_2 may be achieved either by the dry process in which all components are physically bended in dry state or by the wet process in which an aqueous slurry of hydrated TiO_2 is used to uniformly disperse other components around each hydrated TiO_2 particle. Advantageously, the above components are added to a hydrated TiO_2 cake free from various impurities produced in a commercial TiO_2 pigment plant as an intermediate product, if necessary after dispersing the cake in an aqueous

medium, and then the mixture is thoroughly stirred. The resulting mixture containing the aluminum, zinc and potassium additives is then dehydrated to a hydrated TiO_2 content from 50 to 65% by weight prior to calcination at a temperature from 900 to 1100℃ which is conventionally employed in the commercial TiO_2 pigment plant. When the calcination temperature is lower than the above range, the primary particles do not sufficiently grow to the desired size resulting in decreased IR shielding effect. Conversely, when the calcination temperature is higher than the above range, milling of the product into fine particles will become difficult due to excessive fusion or sintering also resulting in decreased IR shielding effect.

[0024]

The TiO_2 particles of the present invention may optionally be coated with an amount of inorganic or organic coating materials sufficient to improve dispersibility, electrical property or weatherability necessary for incorporating to paint formulations or plastic molding compounds. Inorganic coating material may be those conventionally employed for coating TiO_2 pigments. Examples thereof are oxides or hydrated oxides of Al, Si, Zr, Zn, Ti, Sn, Sb or Ce.

The oxide or hydrated oxide coating material may be formed in situ from, for example, sodium aluminate, aluminum sulfate, sodium silicate, hydrated silicic acid, zirconium sulfate, zirconium chloride, zinc sulfate, zinc chloride, titanyl sulfate, titanyl chloride, tin sulfate, tin chloride, antimony chloride, cerium chloride or cerium sulfate. Examples of organic materials includes aminosilanes, alkylsilanes, polyether silicone, silicone oil, stearic acid, magnesium stearate, zinc stearate, sodium stearate, lauric acid, alginic acid, sodium alginate, triethanolamine, or trimethylolpropane. The above coating material may be used in combination, and the species and amount thereof may be selected depending upon particular useage and desired properties.

[0025]

The TiO_2 particles thus produced may be incorporated into paints, printing inks, plastic molding compounds or cosmetic preparations in order to impart with IR shielding effect.

[0026]

For use in paint or printing ink formulations, the amount of TiO_2 particles of the present invention to be added may vary depending upon particular

applications and generally ranges 1 to 500 weight parts per 100 weight parts of vehicle resin. Examples of the vehicle resins are acrylic-melamine, air-drying acrylic, acrylic-urethane, polyester-melamine, alkyd-melamine, polyurethane, nitrocellulose, fluoro, and vinyl chloride-vinyl acetate copolymer resins. The paint or printing ink formations may contain other pigments. Examples thereof include flaky pigments such as mica, sericite or talc, inorganic pigments such as calcium carbonate, barium sulfate, silica balloons, zirconium oxide, TiO_2 pigment, TiO_2 UV blocker, or zinc oxide, metal flakes such as aluminum flake, and inorganic or organic color pigments and dyes having a high transmission or reflectivity to IR wave range of the solar radiation. The TiO_2 particles of the present invention may be incorporated into paint or printing ink formulations as a suspension in water or organic solvent such as hydrocarbons, alcohols, ethers, esters, ester-alcohols or ketones. The mixture is then processed in a conventional apparatus such as paint conditioner, disper or sand grind mill to produce a uniform dispersion. The resulting formulation may be applied onto a metallic or plastic substrate using bar coater, brush, air spray gun or static coating

machine to a desired film thickness. The coating film is then baked, depending upon the type of vehicle resin, at a temperature between 100℃ and 180℃ for a period of time between 10 minutes and 40 minutes.

[0027]

For use in plastic molding compounds, the TiO_2 particles of the present invention are blended with a thermoplastic resin such as polyolefin, polystyrene, polyethylene terephthalate, or polyvinyl chloride. The amount of TiO_2 particles may vary depending upon particular applications of the product and generally ranges between 0.2 and 50 weight parts per 100 weight parts of the resin. The plastic molding compound may contain a lubricant, antioxidant or heat stabilizer. Examples thereof include zinc stearate, calcium stearate, aluminum stearate, magnesium stearate, zirconium stearate, calcium palmitate, sodium laurate and other fatty acid metal salts. These additives are preferably incorporated in an amount from 0.01 to 5% by weight of the plastic molding compound. The molding compound may optionally contain flaky pigment such as mica, sericite or talc, inorganic pigments such as calcium carbonate, barium sulfate, silica balloons, zirconium oxide, TiO_2 pigments, TiO_2 UV blocker, or zinc oxide,

metal flakes such as aluminum flake, and inorganic or organic color pigments and dyes having a high transmission or reflectivity to IR wage range of the solar radiation. The TiO_2 particles may be blended with the resin by mixing them in a mixer such as tumbler or Henschel mixer in dry state and then kneading the mixture in molten state in Bunbury mixer, hot roll mill, extruder or injection molding machine.

[0028]

Because the TiO_2 particles of the present invention have a primary particle size as large as from 0.5 to 2.0 μ m, they have not only higher IR shielding effect but spreadability in comparison with known TiO_2 particles. The term "higher spreadability" as used herein refers to lower stationary and rolling friction coefficients against human skin. Therefore, the TiO_2 particles of the present invention may be added to foundational cosmetic preparations such as pressed powder foundation, powder foundation or liquid foundation, or makeup cosmetics such as face color, lip stick or rouge in order to improve spreadability. The TiO_2 particles may be incorporated in an amount of 1 to 50%. The cosmetic preparations or compositions may

contain solid or semi-solid oil components such as vaseline, lanolin, sericin, microcrystalline wax, carnauba wax, candle wax, higher fatty acids or higher fatty alcohols, and/or fluid oil components such as squalane, paraffin oil, ester oil, diglyceride, triglyceride or silicone oil. Other components which are optionally added include hydrophilic or lipophilic polymers, surfactants, ethanol, preservatives, antioxidants, thickening agents, pH adjusting agents, perfumes, UV aborbers, moisturizers, blood circulation enhancers, frigidizers, astringents, disinfectants, and skin activators, Such components may be used to the extent of not adversely affecting the TiO_2 particles of the present invention. The cosmetic composition may contain conventional powder components. Examples thereof includes body pigments such as talc, kaoline, sericite, mica, magnesium carbonate, calcium carbonate, aluminum silicate, magnesium aluminosilicate, calcium silicate or anhydrous silicic acid; inorganic color pigments such as red iron oxide, black iron oxide, yellow iron oxide, ultramarine blue, prussian blue or carbon black; pearl pigments such as TiO_2-mica, iron oxide-mica or bismuth oxychloride; dyes such as tar or natural dyes; organic

powders such as nylon powder, silicone powder, polyethylene or polypropylene powder, silk powder or crystalline cellulose, and inorganic UV blockers such as TiO_2 microparticles, ZnO microparticles or cerium oxide microparticles. Such powders may be surface-treated with fluorine compounds, silicone, metallic soap, wax, oil and fats, hydrocarbons or a combination thereof. The powder components may be used in conjuction with resins, oils, organic solvents, water or alcohols.

[0029]

In order to incorporate the TiO_2 particles into cosmetic formulations, the particles may be surface-treated to improve their dispersibility in oily components or to give water-repellecy to the cosmetic formulation. The surface-treatment may be carried out by using known treating agents and known methods. Preferable treating agents are silicones such as methylhydrogenpolysiloxane and aluminum stearate. These agents may be used in combination with UV absorbers, surfactants or thickening agents. The treating method may be dry mixing in a mixer such as Henschel mixer or wet process in an organic solvent.

[0030]

Example

The invention is further illustrated by the following Examples and Comparative Examples without limiting the invention thereto. All percentage and part are by weight unless otherwise indicated.

[0031]

Example 1

TiO_2 particles having a primary particle size of 1.0 μm were produced by the following procedure.

[0032]

A solution of titanyl sulfate was prepared by digesting ilmenite ore with hot concentrated sulfuric acid followed by removing impurities. The resulting solution was thermally hydrolyzed to obtain hydrated TiO_2 as a crude cake which was thoroughly washed with water to remove any electrolyte. To the purified cake was added an amount of a solution of aluminum sulfate corresponding to 0.2% calculated as Al_2O_3 relative to the TiO_2 content of the cake and the mixture was stirred for 15 minutes. To the mixture were added a solution of potassium hydroxide and a solution of zinc oxide successively in amounts 0.4% calculated as K_2CO_3 and 0.4% calculated as ZnO, respectively relative to the TiO_2

content of the cake followed by stirring for 15 minutes after each addition. Then the mixture was dried in a dryer at 110℃ for 7 hours to obtain dry mixture having a TiO_2 content of about 60%. After drying, the mixture was calcined at 950℃ for 2 hours. The calcined product was then pulverized in dry state in a sample mill and finely divided in wet state in a sand grinder mill to obtain an aqueous slurry having a TiO_2 content of about 24-29%. To the slurry was added an amount of sodium aluminate corresponding to 2.0% calculated as Al_2O_2 relative to the TiO_2 content followed by neutralization with sulfuric acid. Then the treated particles was collected by filtration, washed with water, and dried in a dryer at 110℃ for 12 hours. Finally the dried product was divided into finer particles in a liquid energy mill.

[0033]

A photograph of the resulting TiO_2 particle was taken using a transmission electron microscope (Jeol Ltd., model JEM-1230) and the volume average diameter was determined by measuring the diameter along the X-axis that divides the image of particles into equiareal halves (Martin's diameter) using an automated image analyzer (NIRECO, model LUZEX

AP). The size of primary particles was about 1.0 μm.

[0034]

Preparation of coating composition

A commercial clear acrylic lacquer and the above TiO_2 particle were weighed into a plastic mayonnaise bottle in 100 parts each as solids. After closing the bottle with a cap, the content was dispersed for 1 hour using a paint conditioner.

[0035]

Preparation of test specimen

The above coating composition was applied onto a PET film to a dry film thickness of 5 μm using a automated bar coater, set for 10 minutes, and baked at 140℃ for 30 minutes.

[0036]

Example 2

The procedure of Example 1 for preparing TiO_2 particles, coating composition and test specimen was followed except that the calcination temperature was changed to 980℃. The primary particle size determined by the same way as in Example 1 was 1.2 μm.

[0037]

Example 3

The procedure of Example 1 for preparing TiO_2

particles, coating composition and test specimen was followed except that the calcination temperature was changed to 1020℃. The primary particle size determined by the same way as in Example 1 was 1.5 μm.

[0038]

Example 4

The procedure of Example 1 for preparing coating composition and test specimen was followed except that the amount of the same TiO_2 particle in the coating composition was changed to 50 parts.

[0039]

Comparative Example 1

The procedure of Example 1 for preparing coating composition and test specimen was followed using a commercial TiO_2 pigment (Tayca Corporation, JR-701) instead of TiO_2 particles produced in Example 1. The primary particle size of the pigment determined by the same way as in Example 1 was 0.27 μm.

[0040]

Comparative Example 2

The procedure of Example 1 for preparing coating composition and test specimen was followed by using zinc titanate particles instead of TiO_2

particle produced in Example 1.

[0041]

The zinc titanate was produced as follows. The hydrated TiO_2 produced in Example 1 was mixed solely with an amount of zinc oxide corresponding to 200% calculated as ZnO relative to the TiO_2 content of hydrated TiO_2 oxide. The mixture was stirred for 15 minutes, dried to a TiO_2 content of 50-65% and calcined at 1000℃ for 2 hours. After calcination, the product was processed in the same way as in Example 1. The calcined product was identified as zinc titanate using an X-ray diffractometer (Phillips, X'Pert Pro). The primary particle size of zinc titanate determined in the same way as in Example 1 was 1.0 μm.

[0042]

Comparative Example 3

The procedure of Comparative Example 1 for preparing coating composition and test specimen was followed except that the amount of TiO_2 pigment in the coating composition was changed to 50 parts.

[0043]

Comparative Example 4

The procedure of Comparative Example 1 for

preparing coating composition and test specimen was followed except that the amount of TiO_2 pigment was changed to 40 parts.

[0044]

Measurement of IR transmission

IR transmission was determined for test specimens containing TiO_2 at 50% level (Examples 1 and 2, Comparative Examples 1 and 2) using FT IR spectrophotometer (NIRECO, PRTGE 60) in the wavelength range from 0.7 to 3 μm. The transmission curve of each specimen is shown in Figs. 1-4.

[0045]

The integrated IR transmission value over wavelength range between 1.4 and 3.0 μm was calculated from the curve of Figs. 1-4. Percent transmission is represented by the following equation and shown in Table 1 below.

$$\%\ \text{transmission} = \frac{\text{integrated value of specimen}}{\text{integrated value of blank}} \times 100$$

[0046]

Table 1

Specimen	% IR transmission
Example 1	5
Example 2	3
Comp.Exm.1	36
Comp.Exm.2	27
PET film	95

[0047]

As shown in transmission curves of Figs. 1-4 and Table 1 above, the TiO_2 particles of the present invention exhibit remarkably lower IR transmission and higher shielding in the wavelength range from 1.4 to 3.0 μm compared to commercial TiO_2 pigment or zinc titanate particles.

[0048]

Thermal IR shielding test

A small window of 40mm x 50mm size was cut on the top face of foamed polystyrene box and the window was closed with the film prepared in

Examples 1-4 and Comparative Examples 1-4, respectively. The interior of the box was initially kept at room temperature (23℃). Then an IR lamp was placed at a distance of 15cm above the window and turned on for 20 minutes to irradiate the interior of the box with IR radiation. The inner temperature of the box was monitored each time and temperature differencial was determined at the end of irradiation by subtracting the inner temperature when irradiating through the film prepared in Examples and Comparative Examples from the inner temperature when irradiating through the control PET film not having any coating. The results are shown in Table 2 below.

[0049]

Table 2

Specimen	TiO_2 content	Inner Temp.	Temp.Diff.
Example 1	100 wt parts	44℃	30℃
Example 2	"	42℃	32℃
Example 3	"	43℃	31℃
Comp.Ex.1	"	51℃	23℃
Comp.Ex.2	"	51℃	23℃
Example 4	50 wt. parts	48℃	26℃
Comp.Ex.3	"	56℃	18℃

Comp.Ex.4	"	58℃	16℃
Control(PET)	None	74℃	—

[0050]

As demonstrated in Table 2, the TiO_2 particles of the present invention retard elevation of temperature by shielding thermal IR radiation.

[0051]

Example 5

Thermal IR shielding of TiO_2 particles of the present invention was evaluated in plastic molding compounds.

[0052]

The TiO_2 particles produced in Example 2 were mixed with polyethylene in a proportion of 0.5 parts by weight relative to 100 parts by weight of polyethylene. The mixture was kneaded using a pair of hot rolls and pressed into a sheet having a thickness of 100 μm.

[0053]

Comparative Example 5

Example 5 was followed using commercial TiO_2 pigment (Tayca, JR-701) instead of the TiO_2 particles of Example 2.

[0054]

The thermal IR shielding test was repeated for the polyethylene sheets of Example 5 and Comparative Example 5 to determine temperature differential from the inner temperature of the box. The results are shown in Table 3 below. The temperature differential in Table 3 refers to the inner temperature differential between the sheet prepared in Example 5 or Comparative Example 5 and the corresponding polyethylene sheet to which TiO_2 was not added.

[0055]

Table 3

Specimen	TiO_2 content	Inner Temp.	Temp.Diff.
Example 5	0.5 wt.parts	48℃	7℃
Comp.Ex. 5	0.5 wt.parts	51℃	4℃
Control(PE sheet)	None	55℃	—

[0056]

As demonstrated in Table 3, the TiO_2 particles of the present invention retard elevation of temperature by shielding thermal IR radiation when adding to plastic molding compounds.

[0057]

Example 6

Thermal IR shielding of TiO_2 particles of the particles of the present invention was evaluated in cosmetic compositions.

[0058]

Using the TiO_2 particles produced in Example 2, a cosmetic composition was prepared.

[0059]

Formulation

Powder components:	Wt. Parts
TiO_2 of Example 2	20
Mica	36
Sericite	10
Talc	10
Oily components:	
Liquid paraffin	17.5
Isopropyl palmitate	5
Lipophilic glyceryl monooleate	1.5

[0060]

The powder components and oily components were separately mixed together. An antioxidant, preservative and perfume were dissolved q.v. in the oily component mixture. All components were placed in a ribbon blender and mixed well. The mixture was then compressed in a mold.

[0061]

The resulting composition was applied uniformly on the entire surface of a surgical tape (8x5cm) using a finger in an amount of 2mg/cm^2 to prepare a specimen.

[0062]

Comparative Example 6

Example 6 was followed using commercial TiO_2 pigment (Tayca, JR-701) instead of the TiO_2 particles of Example 2 to produce the cosmetic composition and specimen.

[0063]

The thermal IR shielding test as described above was repeated using the specimens prepared in Example 6 and Comparative Example 6. The results are shown in Table 4 below. The temperature differential therein refers to the inner temperature of the box closed with the specimen of Example 6 or Comparative Example 6 subtracted from the inner temperature of the box closed with untreated surgical tape.

[0064]

Table 4

Specimen	TiO_2 content	Inner Temp.	Temp.Diff.

Example 6	20 wt.parts	35℃	26℃
Comp.Ex.6	20 wt.parts	40℃	21℃
Control(surgical tape)	—	61℃	—

[0065]

As demonstrated in Table 4, cosmetic compositions containing the TiO_2 particles of the present invention retard elevation of temperature by shielding thermal IR radiation.

[0066]

Spreadability test of cosmetic compositions

Besides thermal IR shielding, the TiO_2 particles of the present invention can improve the spreadability of cosmetic compositions due to greater particle size than conventional TiO_2 pigment. The following are comparative experiments of the TiO_2 of the present invention and commercial TiO_2 pigment for spreadability.

[0067]

Example 7

The TiO_2 particles produced in Example 1 having a primary particle size of 1.0 μm were surface-treated with dimethylpolysiloxane. Using the surface-treated TiO_2 particles, a pressed powder foundation composition was produced.

[0068]

Formulation

Powder components:	wt. parts
Surface-treated TiO_2	15
Talc	20
Sericite	30
Mica	20
Iron oxide(red,yellow,black)	3
Oily components:	
Lanolin	2.4
Squalane	2.4
Capryloyl capryl triglyceride	1.8
2-Ethylhexanoyl triglyceride	1.8
Methylphenylpolysiloxane	3.6

[0069]

The powder components were mixed in Henschel mixer for 5 minutes. To this were added portionwise the oily components heated to 50-60℃ and mixing was continued for additional 5 minutes. The resulting mixture was transferred in a mold and compressed to obtain the desired product.

[0070]

Example 8

Analoguous to Example 7, a pressed powder

foundation composition was produced using the surface-treated TiO_2 particles of Example 1.

[0071]

Formulation

Powder components:	Wt. parts
Surface-treated TiO_2	10
TiO_2 microparticles(Tayca,MT-100TV)	1
Talc	19
Sericite	30
Mica	18
Anhydrous silicic acid	2.5
Nylon powder	4.5
Iron oxide (red,yellow,black)	3
Oily components:	
Lanolin	2.4
Squalane	2.4
Capryloyl capryl triglyceride	1.8
2-Ethylhexanoyl triglyceride	1.8
Methylphenylpolysiloxane	3.6

[0072]

The powder components were mixed in Henschel mixer for 5 minutes. To this were added portionwise the oily components heated to 50-60℃ and mixing

was continued for additional 5 minutes. The resulting mixture was transferred in a mold and compressed to obtain the desired product.

[0073]

Comparative Example 7

The commercial TiO_2 pigment used in Comparative Example 1 was surface-treated with dimethylpolysiloxane. Using the surface-treated TiO_2 pigment, Example 7 was repeated to produce a pressed powder foundation composition.

[0074]

Application feeling test:

The application feeling of cosmetic compositions such as liquid foundation and powders are affected by the spreadability of powder components incorporated therein.

[0075]

The pressed foundations of Examples 7-8 and Comparative Example 7 were tested for the application feeling by applying the foundation directly to the skin of 10 test panelers. The application feeling sensed by the panelers was evaluated according to the following schedule.

[0076]

<u>Schedule</u>

Very good: 8-10 panelers reported to be not creaky and highly spreadable.

Good: 6-7 panelers reported to be not creaky and highly spreadable.

Fair: 3-5 panelers reported to be not creaky and highly spreadable.

Bad: 0-2 panelers reported to be not creaky and highly spreadable.

[0077]

The results are shown in Table 5 below.

Composition	TiO_2 content	Application feeling
Example 7	15 wt.%	Very good
Example 8	10 wt.%	Very good
Comp.Ex.7	15 wt.%	Bad

[0078]

Reflectivity of Visible Light

TiO_2 particles of Examples 1-2 and commercial TiO_2 pigment used in Comparative Example 1 were each compressed at 100 MPa into tablets. The reflectivity in the wavelength range between 0.4 and 0.8 μ m relative to a standard MgO tablets was measured using a spectrophotometer (Hitachi Ltd.,

Model U-3000). Figs. 5-7 show the reflectivity curves of TiO_2 particles of Examples 1-2 and commercial TiO_2 pigment.

[0079]

The percent reflection was calculated from integrated value of reflectivity curve of Figs. 5-7 according to the following equation.

$$\%\ \text{reflection} = \frac{\text{integrated reflectivity value}}{\text{integrated value of standard}} \times 100$$

[0080]

The results are shown in Table 6 below.

[0081]

Table 6

TiO_2	% Reflection of visible light
Example 1	90.6
Example 2	92.0
Comp.Ex.1	96.4

[0082]

TiO_2 particles having decreased visible light

reflection are advantageous for use in cosmetic compositions because of less whitening and less blueish luminescent effects on the cosmetic composition than TiO_2 pigment.